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SCOPE OF WORK FIELD AND LABORATORY MEDIA TESTING FOR URANIUM SEQUESTRATION IN GROUNDWATER

Nuclear Metals, Inc. Superfund Site Concord, Massachusetts

Prepared for

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TABLE OF CONTENTS

1.	INTRODUCTION	3
1.1	Purpose and Proof-of-Concept.....	3
1.2	Scope of Work Organization	4
2.	RATIONALE FOR SELECTED MEDIA	5
2.1	Apatite.....	5
2.2	Vanadate-Modified Apatite	6
3.	TESTING PROCEDURES	7
3.1	In-Well Column Testing	7
3.1.1	Passive Deployments.....	7
3.1.2	Active Deployments.....	8
3.1.3	Groundwater Sample Collection	9
3.1.4	Canister/Media Collection and Analysis	10
3.1.5	Solids Analysis of Field Media	11
3.2	Laboratory Column Testing.....	11
3.2.1	Media Uptake Testing	11
3.2.2	Solid Phase Analysis	13
3.2.3	Leaching/ Stability Testing	15
4.	PROJECT TEAM	16
5.	REPORTING AND PROJECT SCHEDULE.....	16
6.	REFERENCES	17

LIST OF TABLES

Table 1: Depleted Uranium Plume Monitoring Well Details

Table 2: Depleted Uranium Plume Geochemistry

Table 3: Field Media Testing and Groundwater Monitoring Schedule

Table 4: Soil Lithologies in Screened Intervals in Uranium Plume

Table 5: Proposed Schedule for Media Testing

LIST OF FIGURES

Figure 1: Total Uranium Concentrations in Overburden Groundwater

Figure 2: Depleted Uranium Distribution in Cross Section

Figure 3: In-Well Column Testing Schematic

Figure 4: Thermodynamic Modeling of Uranium (VI) Solid Phase Saturation Indices

Figure 5: Example laboratory column experimental set-up (*SMU Water and Soil Chemistry Laboratory*)

APPENDICES

Appendix A: Geosyntec Low Flow Field Sampling Form and EPA Low Flow Sampling Procedures

1. INTRODUCTION

This Scope of Work (SOW) provides field and laboratory procedures for evaluating the effectiveness of selected media for sequestering dissolved phase uranium in groundwater at the Nuclear Metals, Inc. (NMI) Superfund Site in Concord, Massachusetts.

Depleted uranium (DU) penetrators and bullets were manufactured at the NMI facility for the United States (US) Army. DU billets were delivered to the facility with copper jacketing which was removed using nitric acid. The objective of the process was to completely remove the copper and remove as little DU as possible; however, the process resulted in dissolution of copper and uranium in the nitric acid. Residual sludge from these operations was placed within a topographic depression that is referred to as the Holding Basin. The Holding Basin was actively used from 1958 to September 1985 for disposal of waste sludge, and the nitric acid solution was neutralized with lime. Although uranium sludge in the Holding Basin has been excavated and removed, residual elevated uranium remains in stratified drift soils beneath the Holding Basin footprint. Uranium has migrated in groundwater approximately 350 feet downgradient of the Holding Basin and investigation activities indicate that uranium is sorbed to saturated zone soils immediately beneath and downgradient of the Holding Basin. Uranium in these soils constitutes an ongoing source to groundwater. Detailed discussions of site geochemistry, uranium distribution, and uranium adsorption/desorption experiments on site soils are included in the NMI Remedial Investigation Report (RI) (de maximis, 2012) and Depleted Uranium Fate and Transport Model Report (Geosyntec, 2011, Appendix to the RI).

As of the most recent sampling in 2012, uranium concentrations in groundwater within the DU plume ranged up to approximately 2,730 µg/L. Table 1 provides a list of wells within the uranium plume, including uranium concentrations and well construction details. Figure 1 shows the Holding Basin and the current estimated extent of the DU plume. A conceptual cross section of the DU plume is illustrated in Figure 2, and the DU plume geochemistry is detailed in Table 2.

1.1 Purpose and Proof-of-Concept

The purpose of the proposed work is to evaluate the effectiveness of apatite and vanadate-modified apatite media for sequestering hexavalent uranium via sorption or precipitation of insoluble mineral forms of uranium without manipulation of aquifer geochemistry. Insoluble uranium minerals are relatively permanent in a practical sense (i.e., the dissolution timeframes are very long on a human scale) given stable groundwater geochemistry such as pH, oxidation reduction potential (ORP), dissolved oxygen and alkalinity. This SOW is designed to provide an initial proof-of-concept prior to issuance of the Record of Decision (ROD) for the NMI Site. The proof-of-concept will include simultaneous bench scale and field pilot testing to demonstrate if uranium is in fact sequestered by one or more media and identify the method of sequestration (adsorption and/or mineralization). Simultaneous testing in both the laboratory and field is necessary for the following reasons:

- Field Testing - Given the short duration timeframe to provide proof-of-concept prior to issuance of the ROD, it is necessary to reach a conclusion on

the efficacy of at least one of the proposed media in-situ under existing site conditions with no laboratory controls. Apatite II was chosen for the in-situ testing because it is commercially available and has undergone more testing than the other media to date.

- Laboratory Testing- Evaluating the proposed media in a laboratory setting allows for controlled hydraulics and geochemistry to assess the forms and associated stability of sequestered uranium. Because vanadate coated apatite is a promising media to sequester uranium (i.e., may result in phases that are more insoluble than those associated with apatite II), this media along with Apatite II will be tested in the laboratory prior to synthesizing large scale quantities for field use.

The remedial options identified for the uranium plume include containment of the Holding Basin source; however, residual uranium sorbed to aquifer solids outside of the Holding Basin boundaries is expected to be a continuing source to groundwater (although with a much lower total mass than the Holding Basin). Field scale application of in-situ sequestration using one of the media listed above may be a viable approach to immobilize uranium and reduce concentrations of uranium in groundwater downgradient of the Holding Basin. Media could be emplaced by several methods, but high pressure injection is likely the most feasible.

1.2 Scope of Work Organization

This SOW is organized into the following sections:

Section 1: Introduction

Section 2: Rationale for Selected Media

Section 3: Testing Procedures

Section 4: Project Team

Section 5: Reporting and Project Schedule

Section 6: References

2. RATIONALE FOR SELECTED MEDIA

Based on our evaluation of the state-of-the-technology for in-situ aqueous phase uranium remediation, no in-situ remedies to address uranium in groundwater have been demonstrated at the full field scale. However, success has been demonstrated at the bench and pilot scale for multiple media. There is strong promise for the use of several media for in-situ sequestration of uranium that are well suited for the groundwater geochemistry at the NMI Site. To provide proof-of-concept for an in-situ remedy, we recommend field and laboratory experiments using apatite and vanadate-modified apatite. A discussion of these media is provided below. Apatite and vanadate-coated apatite media were also discussed in the Feasibility Study Report (*de maximis*, 2013).

2.1 Apatite

Many metals and radionuclides dissolved in groundwater can be immobilized through abiotic precipitation as insoluble phosphate minerals. This approach has been applied at the bench and field scale for the sequestration of metals such as Pb, Cd, Zn, Al, Cu, and U and other actinide elements (Bostick et al., 2003; Fuller et al., 2002; Fuller et al., 2003; Krestou and Panias, 2004; Wellman et al., 2008; Simon et al., 2008). There are three possible reaction mechanisms responsible for the removal of uranium from solution using apatite: (1) dissolution of apatite and subsequent precipitation of U(VI)-phosphates such as chernikovite, autunite, or Ba-autunite, (2) sorption, and (3) ion exchange (Simon et al., 2008). These reactions can occur concomitantly, and will vary depending on the groundwater geochemistry. The solubility products for U(VI)-phosphates such as autunite are extremely low (e.g., $K_{sp} = 10^{-49}$; Conca, 2000). In fact, in oxidizing, apatite-rich environments, it has been suggested that the U(VI)-phosphates formed via dissolution-precipitation reactions are stable for tens to hundreds of thousands of years (Jerden and Sinha, 2003).

Apatite is readily available via natural and synthetic sources. Apatite II, produced by Phosphate Induced Metal Stabilization (PIMS) NW, Inc. (<http://www.pimsnw.com/>), is synthesized from fish bones (a waste product from commercial fish processing) and is a very reactive and cost-effective apatite product (Wright et al. 2004). It is formulated to have high internal porosity, be poorly crystalline (but with sufficient nucleation sites for precipitation of metal phosphates), contain minimal trace metals, contain minimal fluorine in the hydroxyl position, and be fully carbonated. The apatite structure is stable over a wide range of pH conditions (2-12), temperature fluctuations (up to 1,000°C), and timescales (millions of years) (Conca, 2000). These properties result in high reactivity and removal efficiency of uranium and other metals from groundwater. Because Apatite II is non-specific for metal adsorption, it is effective at mitigating impacts from multiple metal contaminants.

Demonstration of uranium sequestration from groundwater using apatite has been successfully accomplished in bench and column studies for the Y-12 plant site in Oak Ridge, TN (Matheson, et. al., 2002), and for the 300 Area uranium plume at the Hanford Site in Washington State (Wellman et al., 2008; Szecsody, J.E., et. al., 2012).

2.2 Vanadate-Modified Apatite

Precipitation is an attractive in-situ stabilization mechanism due to the low solubility product of uranyl phosphates. However, uranyl vanadates such as carnotite [$K_2(UO_2)_2(VO_4)_2 \cdot 3H_2O$] have even lower solubility products and thus may be even more favorable for in-situ stabilization (Tokunaga, 2009). Precipitation and stability of uranyl vanadates is favorable under circum-neutral pH conditions (5.5-7) (Tokunaga, 2009). At vanadium concentrations typical of groundwater and over a broad pH range, vanadate occurs primarily as the oxyanion $H_2VO_4^{-1}$, which is similar to the phosphate ion. Moderate levels of vanadium and potassium ($V(V) < 2 \mu M$ and $K^+ < 2 mM$) at these pH levels may result in the removal of uranium from groundwater to concentrations below the MCL via the formation of potassium or calcium vanadate minerals (Tokunaga et al., 2009). The Water and Soil Chemistry (WSC) Lab at Southern Methodist University (SMU) has developed a mixed phase calcium vanadate-phosphate with very high specific surface area. The potential uranium solids formed in association with this material are highly insoluble (Figure 4). The proposed material should, therefore, remove uranium to even lower solution concentrations and yield a more stable product. Further, the end product has a broad pH stability range relative to more traditional non-vandate precipitates as well as sorbed phases (Figure 4).

3. TESTING PROCEDURES

To provide proof-of-concept, field and laboratory experiments will be used to demonstrate the ability of the apatite media to sequester uranium. Sequestration will be quantified by measuring the mass of uranium on the media, relative uptake rates, as well as conversion percentages from sorbed U(VI) to precipitated species (i.e., uranyl phosphates, uranyl vanadates or mixed vanadate-phosphate precipitates). The extent of occlusion will also be evaluated for each medium to evaluate the potential for reductions in hydraulic conductivity over time. The results of these tests will be used to identify the most effective medium and develop the basis for a full scale design.

A successful full-scale application would assure long-term uranium sequestration under the conditions found at the NMI Site with no active operation and maintenance (i.e., monitoring only). The following subsections describe the testing procedures for both in-situ and laboratory tests.

3.1 In-Well Column Testing

In-well column testing will include the deployment of canisters containing Apatite II acquired directly from PIMS. Testing of media in the laboratory will allow for comparisons between the media. The canisters will be installed in four monitoring wells: two as passive deployments and two with active groundwater extraction through the canisters. Design of the passive and active deployments is described below.

3.1.1 Passive Deployments

There are approximately eight wells within the DU plume with uranium concentrations above the MCL of 30 µg/L. Construction details and uranium concentrations for these wells are summarized in Table 1. Estimates of uranium flux through the wellscreens (based on uranium concentration at each well), the hydraulic conductivity of soils in the screened interval and the estimated hydraulic gradients at each well are also presented in Table 1. Two wells were selected for passive deployments that are approximately representative of the range in uranium concentrations, uranium flux through the screens and location within the plume. Passive deployments are proposed for wells HBPZ-2R (uranium = 1,810 µg/L) and MW-S16 (uranium = 105 µg/L). These locations are shown on Figure 1. Proposed Sample IDs and groundwater sampling requirements are presented in Table 3.

The canisters of Apatite II media will be constructed of two-foot lengths of 1.25-inch diameter 20-slot PVC wellscreen or wire mesh material. Each passive deployment will consist of two vertically stacked canisters that will be placed within the well. This will allow retrieval of single canisters at different times. The wellscreen lengths are 10-15 feet. The remaining 6-11 feet of wellscreen not intersected with media canisters will be isolated using a solid piece of 1.5-inch (1.9-inch outside diameter) pipe to encourage preferential flow through the media. The media will be ground to approximately medium to coarse sand size such that the hydraulic conductivity of the media will be at least as high as the surrounding formation as to not limit migration through the media. A review of soil lithology within the screened interval at HB-PZ-2R and

MW-S16 was conducted to target the deployment of the canisters in the depth intervals anticipated to have more permeable sub-strata. Soil lithologies for wells in the uranium plume are presented on Table 4. Based on this review the following depths have been selected for passive deployments:

Passive Deployment Well Target Depths

Well ID	Screened Interval Depth (ft bgs)	Media Deployment Depth (ft bgs)	Blank Section Depth (ft bgs)
HBPZ-2R	36-51	40-44	36-40, 44-51
MW-S16	69-79	75-79	69-75

3.1.2 Active Deployments

In order to control and augment the mass of uranium migrating across the media, two wells were selected to be tested using media canisters through which groundwater will be actively pumped. A down-well, low-flow submersible pump will be used in each well to pump groundwater through two separate media canisters in parallel located in the same well. It is anticipated that the pumping rate through the media will be approximately 0.1 liters/minute. A third line will be installed in each well to sample influent groundwater without passing through the Apatite II. Above ground at each well, a larger diameter media canister (approximately 4-inch diameter) will be installed along this third line for a supplemental/backup treatment evaluation. These larger diameter canisters will have a lower water velocity and higher retention time through the media compared to the smaller diameter in-well canisters.

Use of an accelerated flow rate through the media at two locations will result in more pore volume exchanges in a shorter timeframe than will occur in the passive deployments, thus simulating treatment over a longer timescale. Two wells were selected to receive active deployments that are approximately representative of the range in uranium concentration and location within the plume. Active deployments are proposed for wells MW-S24 (uranium = 2,730 µg/L) and MW-8A (uranium = 447 µg/L). Proposed Sample IDs and groundwater sampling requirements are presented in Table 3. The locations are presented on Figure 1.

A schematic of the active deployment apparatus is presented as Figure 3. This figure shows the in-well pump, tubing and media configuration, and the ex-situ testing apparatus. Extracted water that is pumped through the media will be controlled and monitored using flow meters, totalizers and valves. Flow volumes, together with periodic analysis of influent and effluent and analysis of the media, will allow for quantification of the uranium mass balance and treatment efficiency. Sampling over time may also provide relevant breakthrough information, although breakthrough is not anticipated to occur during the proposed testing. Groundwater will be sampled according to low flow protocols as described in Section 3.1.3.

Extracted groundwater will be stored on-site for later disposal according to procedures presented in the Site Quality Assurance Project Plan (QAPP) (*de maximis*, 2004).

3.1.3 Groundwater Sample Collection

Groundwater in wells at the passive canister locations will be sampled prior to media deployment using low-flow techniques in accordance with USEPA guidance (USEPA, 2010) using a variable speed submersible pump (Appendix A). For low flow sampling, the purge rate will be low enough to avoid excessive drawdown in the well (< 0.3 ft) while drawing formation water horizontally from the target depth into the pump. These wells have typically been sampled at flow rates between 0.1 and 0.5 L/min. The flow rate will be adjusted based on continuous monitoring of the drawdown of the water level in the well. Once sampled, the low flow pump will be removed and the passive canisters installed. Groundwater in the wells slated for passive canisters will also be sampled periodically (see Table 3) and at the time of media extraction.

The pump intake will be positioned in the portion of the screen targeted for media deployment and purging will continue until measured water quality parameters including temperature, pH, specific conductivity, dissolved oxygen (DO), ORP, and turbidity are stable. Water quality parameters will be recorded every five minutes and considered stable when three consecutive readings are within percentage requirements presented in the standard operating guideline for low flow sampling (Appendix A). Field parameters except turbidity will be monitored using a YSI 650XL[™] multi-parameter meter with flow-through cell (or equivalent). DO will be recorded as concentration in mg/L as opposed to the percent theoretical DO based on temperature. Turbidity will be measured by collecting grab samples while purging each well. Each grab sample will be analyzed using a LaMotte 2020 turbidity meter or equivalent. The YSI multi-meter will be calibrated prior to use. Low flow sampling procedures, forms and instrument calibration criteria and corrective actions for the YSI and LaMotte instruments are presented in Appendix A.

For the active well canister locations, a low flow pump will already be installed in the wells and the pump will be continuously active. The non-treated line from the pump will be run through a low flow cell with a multi-meter probe at the ground surface. Groundwater from these wells will be sampled at the initial deployment time and then periodically during the testing period as shown on Table 3. Geochemical field data will be collected at the time of each groundwater sampling event. Sample ports on each of the three separate lines in the active setup (media canister 1, media canister 2 and untreated water) will allow the low flow cell to easily be attached to each stream for geochemical data collection prior to sample collection. Groundwater that has passed through the large canisters will not be analyzed.

Groundwater samples will be collected into certified pre-cleaned, pre-preserved bottles by disconnecting the tubing from the flow through cell and directing the flow into the bottles. Samples will be collected for the following parameters:

- Total and Dissolved Metals and Cations (uranium, iron, calcium, potassium, sodium) by EPA Method 6020A and vanadium by EPA Method 6010;
- Total phosphorous by EPA Method 365.4, and;
- TOC and TIC by EPA Method 9060.

Samples collected for metals analysis will be collected for both total (unfiltered) and dissolved (filtered) analysis. Filtered samples will be collected through an in-line 0.45 µm filter. The analytes will be tested only from the open line prior to passing through the above ground canisters.

The sample pump and water level meter used in the passive deployment wells will be decontaminated after each use according to decontamination procedures presented in the Site QAPP (*de maximis*, 2004). The QAPP calls for duplicate groundwater samples at a rate of 1 per 20 for each analyte and an equipment blank at a rate of one per analyte per sampling round. As shown in Table 3, we will collect samples every other day for the first week at both passive and one active location, and then once per week through week four. Therefore, we will take duplicate samples on day seven of the first week, and then at week ten for one of the active deployments. Because this work includes numerous sample rounds at only four locations, an equipment blank per sampling event is not practical. We will collect one equipment blank at the beginning of the testing procedures and then another at week four. All samples will be analyzed by the project laboratory, GEL of Charleston, SC.

3.1.4 Canister/Media Collection and Analysis

One canister from each well will be recovered at approximately 30 and 90 days after deployment. After the first passive canister extraction, a 2-foot solid riser will be installed to replace the location of the first media canister. Because the extracted canister from the passive location is composed of open wellscreen or wire mesh, it will be packed in solid 2-inch diameter PVC filled with site groundwater and capped on each end. The 2-inch diameter (2.37-inch OD) PVC will be placed into 4-inch diameter solid PVC and capped on each end, with crushed ice filling the annular space. The extracted media canisters from the active locations are solid walled and will only need to be sealed on each end and placed on ice in a cooler prior to shipment. The extracted media canister packages will be shipped to the WSC Lab at SMU in Dallas, Texas. Analytical testing of this media will include analysis for uranium sorption and mineralization. Details concerning these analyses are presented in Section 3.2.2.

In the unlikely event that solid phase analysis of Apatite II from the active canisters does not indicate sorbed uranium and uranium mineral phases, then the larger diameter back up canisters

will be retrieved, transported in a manner similar to the in-well canisters and then shipped to SMU and analyzed as presented in Section 3.1.5.

3.1.5 Solids Analysis of Field Media

Analysis of retrieved in-well Apatite II media will be conducted to evaluate the mass and nature of sequestered uranium. Samples will be collected and processed according to the Field Media Testing Schedule (Table 5). These analyses will include surface area analysis, bulk X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Sequential Extraction Procedures (SEP) with elemental analysis by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) (see Section 3.2.2). The data from these analyses will be used to estimate the mass of sorbed uranium as well as uranium mineral phases, their crystallinities, morphologies, purities and their changes over time. Each analytical technique will provide one or more key pieces of data as described in Section 3.2.2. If necessary, solid phase analyses of the larger diameter out of well canisters will include a subset of the full suite of analyses presented in Section 3.2.2 depending on results from the in-well media analyses. For example, if results from the in-well media indicate sorbed uranium and not mineralized uranium, then selected solid phase analyses targeted at evaluating mineral phases (such as SEM and TEM) will be completed. Understanding what uranium products have been formed and how they change over the time period of the testing will give insight to the stability of an in-situ remedy using Apatite II.

3.2 Laboratory Column Testing

Laboratory procedures for evaluating uranium uptake on fresh media, solid phase and dissolved phase analyses, and leachability testing of media with sorbed and mineralized uranium are presented below.

3.2.1 Media Uptake Testing

Laboratory column testing will consist of flowing synthetic groundwater through the two types of media under consideration (Apatite II and vanadate-modified apatite) and analyzing both the solids formed and the solution leaving the media. Testing of each medium will be completed by flowing Site groundwater through freshly packed media columns. Groundwater will be collected from well HBPZ-2R using low-flow sampling techniques and shipped overnight to SMU in ice-filled coolers. This testing will provide a more controlled environment than can be achieved in the field to test uranium uptake in each medium. Analysis for uranium uptake on these media will include six (6) columns (2 media for uptake - all in triplicate) with 40 sample collection events per column. While replicate column experiment durations may vary in length for the purpose of solids harvesting the typical length will be 30 days. Sampling will be spread with a front loading to capture faster kinetic processes. Specifically, four samples will be taken on day one, with two samples taken each day from days two through six and then samples will be taken daily. A baseline control will include a column filled with clean, lab grade silica sand only.

During the column testing, influent pressure will be monitored over time to check for occlusion. Additionally, solution chemistry will be coupled with stoichiometry and phase density to back-

calculate volume changes once the precipitating phase is identified. Calculated volumetric changes due to precipitation against time will be compared with the pressure versus time plot to help interpret results.

The range in uranium concentration of groundwater within the uranium plume is approximately 37 to 2,730 µg/L with an average concentration of 754 µg/L. The most recent analyzed uranium concentration at the groundwater source well, HBPZ-2R, was 1810 µg/L. The inlet solution will be placed in an acid washed Nalgene container that will be kept sealed and continuously mixed on a stir plate (Figure 5) throughout the laboratory column testing program. The inlet solution will be pumped using a low-flow peristaltic pump through media packed columns (1 cm ID x 10 cm long) with end packings of clean, lab grade silica sand. The initial flow rates will be 0.1 mL/min; however, the flow rates may be varied based on early results. This initial flow rate will yield approximately two pore volume exchanges per hour within the reactive media. Column effluent will be collected using an auto fraction collector (Figure 5) which will collect samples in acid washed, pre-acidified vials over a set time.

An example of one column setup is illustrated in Figure 5. These experiments will provide detailed solid phase analytical data as well as high resolution solution data directly linked (spatially) to each medium. The solution will be analyzed for the following:

Major analytes: Ca, Mg, K, Na, U, HPO_4^{2-} , HCO_3^- , and Fe.

Trace analytes: Li, Be, Al, V, Cr, Mn, Co, Ni, Cu, Zn, Ga, As, Se, Rb, Sr, Mo, Ag, Cd, Cs, Ba, Pb, Th.

Inductively coupled plasma-mass spectrometry (ICP-MS) will be used to determine dissolved metals in solution. Analyses will be conducted on a Thermo XSeries 2 ICP-MS (Thermo Fisher Scientific, Waltham, MA) in collision cell mode with kinetic energy dispersion. Prior to analysis, an aliquot of samples will be diluted as needed (degree to be determined in the lab) to accommodate the potential range of analyte concentrations. If needed, replicate analyses with adjusted dilution will be employed for this study. Detection limits for uranium using the Thermo XSeries 2 ICP-MS, are in the ng/L range, and even with dilutions (if necessary), the quantification limits will be well below levels needed to understand the influent and effluent uranium concentrations during column testing.

Calibration standards ranging from 0.05 ppb to 500 ppb will be prepared from a multi-element standard that includes all elements of interest. A 5% HNO_3 blank will also be analyzed every 10 samples. Ultrapure 18 MΩ water (Barnstead Nanopure, Fisher Scientific, Pittsburgh, PA) and concentrated, trace metal grade HNO_3 (Fisher Scientific, Pittsburgh, PA) will be used to prepare the 5% HNO_3 solution for dilutions, calibration standards, blanks, and quality control samples.

A potassium bromide tracer will be added to the Site-collected groundwater (5 mg/L Br^-), as bromide is conservative and unreactive with the proposed column materials. Breakthrough curves and effective pore volumes for each column will be calculated based on analyses of bromide in the column effluent.

Column experiments for uranium sequestration will track uranium breakthrough via ICP-MS analysis of U in the effluent (as detailed above). It is expected that both media will have some degree of immediate uptake, likely due to sorption processes. Uptake column testing will continue until significant deviation from the breakthrough curve is observed. Such deviations will signify uranium phase changes within the column (such as sorbed to precipitate).

3.2.2 Solid Phase Analysis

A portion of the media from the uptake columns will be sacrificed for solid phase analyses. Solids will be harvested from replicate columns at various time points. Leaching columns will be replicates of the reactive columns sacrificed for solids analysis and will, therefore, not be affected by solids harvesting. Leaching/stability tests are described in Section 0. The table below presents the solid phase analyses that will provide data to quantitatively evaluate uranium retention with time.

Solid Phase Analyses

Analysis Method		Purpose	Utility
General Media Characteristics			
1	Surface Area Analysis	Surface area per mass analysis (pre- and post- deployment)	Tracking the change in surface area will help address long term viability of reactions.
2	Bulk X-ray Fluorescence (XRF)	Solid phase analysis of U, Fe, and V concentrations	Stoichiometry (paired with structural analysis below) will help identify phases that will impact long-term stability.
Relative Stability of Sequestered Uranium			
3	Sequential Extraction Procedures (SEP), w/ elemental analysis by Inductively Coupled Plasma – Mass Spectrometer (ICP-MS)	Phase association on media post deployment as well as stoichiometry of precipitates formed.	To determine relative abundance of uranium associated with each soil fraction (exchangeable, weakly bound, strongly bound, recalcitrant). A negative result would be the absence of uranium in a strongly bound media fraction, however, SEPs are generally qualitative due to the methods used to extract uranium in each fraction.
Determination of Crystalline vs. Amorphous Phases			
4	X-ray Diffraction (XRD)	Crystallographic analysis of media to assess if high concentrations of uranium precipitates formed on media and, if so, what phases are present	Structural analysis will identify phase. This is necessary for predicting stability. A negative result would indicate the absence of crystalline phases.
5	Electron Microscopy- Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM)	Grain scale (SEM) and subgrain-scale (TEM) analysis to indicate location and morphology of precipitates.	These microscopy techniques will assist in identifying phase by visual observation of media and newly formed phases. A negative result would indicate the absence of crystalline phases.
6	TEM Selected Area Electron Diffraction	Structural analysis of specific grain areas (from extracted media) to identify crystallographic structure of precipitates.	Evaluate selected areas of grain to discriminate between nanocrystalline vs. amorphous phases. A negative result would indicate the absence of crystalline phases.
Grain and Subgrain Elemental Analyses of Sequestered Uranium			
7	SEM Selected Area Energy Dispersive Spectroscopy (EDS)	Elemental analysis at the grain and subgrain-scale. Stoichiometry can indicate the types of precipitates formed.	Identification of elements displayed in SEM and determination of relative proportions of those elements. A negative result would indicate the absence of uranium mineral precipitates.

The SEP analysis is a 5-step process as shown in the table below. The steps include magnesium chloride (MgCl_2); sodium acetate (CH_3COONa , 99%, Mallinckrodt Chemicals); a mix of sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$, Flinn Scientific), sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$, 91%, J.T. Baker) and NaHCO_3 ; dilute HCl ; and ACS grade concentrated nitric acid (HNO_3 , 70%, Mallinckrodt Chemicals). These steps will be conducted in triplicate and reacted in 50-ml centrifuge tubes with an initial starting mass of 200 mg. Samples will be vortexed for a few seconds and then rotated by Labquake Rotators. After reaction completion for each extraction step, the tubes will be centrifuged at 3200 rpm for 15 minutes. An aliquot of supernatant will be taken and filtered through a $0.2\mu\text{m}$ PTFE filter, except for the final concentrated HNO_3 extraction. Each sample will be preserved with nitric acid except for the HCl and HNO_3 extractions. Any remaining available supernatant will be removed and wasted. A rinse of at least 5 mL of 1 M MgCl_2 , followed in succession by 5 mL of nanopure H_2O will be mixed with the sample, vortexed, centrifuged and the maximum possible supernatant decanted prior to addition of the next extractant.

The step by step SEP Method (adapted from Ruttenberg 1992), is presented below.

Step	Extractant	Concentration (M)	Volume (mL)	Reaction Time (hrs)
1	MgCl_2 (pH 8)	1.0	10	1
2	Na-acetate	1.0	10	6
3	Na_3 -citrate	0.30	9	8
	NaHCO_3	1.0	1	
	$\text{Na}_2\text{S}_2\text{O}_4$	0.225 grams		
4	HCl	1.0 M	10	16
5	HNO_3	70%	10	24

3.2.3 Leaching/ Stability Testing

Bench-scale leaching/stability tests will be conducted to evaluate the long-term stability of sorbed/precipitated uranyl species. Both media types will be tested in triplicate for a total of six leaching columns. Completed uptake columns will be used for leaching column tests. The well-characterized media from the replicate uptake columns will be used as starting material in the packed column leach tests. Influent water will match the Site groundwater used in uptake experiments with no uranium added. Experimental setups for these columns will be identical to the uptake columns described above and illustrated in Figure 5.

4. PROJECT TEAM

Geosyntec's project team has many years of experience at the NMI Site. Geosyntec personnel and their roles on the project, as well as key staff at SMU, are listed below.

Geosyntec

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5. REPORTING AND PROJECT SCHEDULE

The proposed schedule for media testing is presented in Table 5. This schedule is designed to provide an initial proof-of-concept prior to the pending ROD. It is anticipated that a Technical Memorandum demonstrating proof-of-concept will be submitted in the February-March 2014 timeframe. However, ongoing field and laboratory bench scale testing will continue until approximately October 2014 to collect additional data to support full scale design and adequately characterize the geochemical mechanisms controlling uranium immobilization and the permanence of the uranyl-species formed. A full report on the field and laboratory uranium sequestration testing will be prepared after these tests are complete and the data has been analyzed.

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TABLES

Table 1
 Depleted Uranium Plume Monitoring Well Details
 Nuclear Metals Superfund Site
 Concord, MA

Well	Uranium Concentration in Groundwater ($\mu\text{g/L}$)	Depth to Water Table (ft)	Depth to Screen Top (ft)	Depth to Screen Bottom (ft)	Ground Surface Elevation (ft)	Screen Interval Hydraulic Conductivity (K) (ft/day)	Hydraulic Gradient (i)	Seepage Velocity (ft/d)	Groundwater Flux Through Screen (est.) (ft^3/day)	Groundwater Flux Through Screen (est.) (L/day)	Uranium Flux Across Screen (mg/day)
MW-S24	2730	50	54	64	186	57.2	0.003	0.9	0.29	8.13	22.19
HBPZ-2R	1810.0	40	36	51	176	60.2	0.003	0.9	0.30	8.55	15.48
HB-12	681.4	48	45	55	183	19.6	0.003	0.3	0.10	2.78	1.90
MW-8A	446.9	58	60	70	192	54.4	0.003	0.8	0.27	7.74	3.46
MW-S02	120.3	59	57	67	192	6.9	0.003	0.1	0.03	0.98	0.12
MW-S16	105.2	54	69	79	188	3.2	0.003	0.05	0.02	0.46	0.05
GZW-7-1	103.2	57	68	78	195	14.8	0.003	0.2	0.07	2.10	0.22
MW-T24	37.3	53	86	90	186	0.4	0.003	0.0	0.00	0.06	0.00

Notes:

1. All wells are 2-inch (0.17 ft) diameter, with a screen area of 1.67 ft^2 .
2. An effective porosity of 0.2 was assumed for the seepage velocity estimate.
3. Uranium concentration data based on groundwater sampling conducted in October 2012.
4. indicates location for passive in-well testing.
5. indicates location for active in-well testing.

Table 2
Depleted Uranium Plume Geochemistry
 Nuclear Metals Superfund Site
 Concord, MA

Analyte		All Locations		GZW-7S			HB-12			HBPZ-2R			MW-8A			MW-S16			MW-S24		
		Total Count	Average	Most Recent	Count	Average	Most Recent	Count	Average	Most Recent	Count	Average	Most Recent	Count	Average	Most Recent	Count	Average	Most Recent	Count	Average
Alkalinity, Calcium Carbonate	µg/L	29	26,284	9130B	2	15165	40300	5	42330	29800	5	26600	20600	7	19721.4	23500	5	20880	40800	5	28964
Calcium	µg/L	26	16,331	16700	2	17550	16600	5	15520	11600	4	13450	16100J	6	13633	15600	4	21125	24800	5	18360
Iron	µg/L	26	193	<120	2	51	118	5	81	54.9	4	43	95.4J	6	53	166	4	812	132	5	155
Nitrate	µg/L	18	2,049	1570	2	1530	<2500	3	2090	<3130	3	2458	1530	4	1723	1280	3	1347	3070	3	3080
Phosphate	µg/L	6	39	53J	1	53	70J	1	70	56J	1	56	<38	1	19	<38	1	19	<38	1	19
Silica	µg/L	3	14,633		0			0			0		15000	1	15000	14500	1	14500	14400	1	14400
Sulfate	µg/L	22	16,650	11000	2	11500	23200	3	20550	20700	3	16850	12400	6	12417	14400	4	13050	22800	4	26100
Uranium (total)	µg/L	56	1,439	122N	3	121	682	11	993	1810J	11	1461	447	12	718	105	8	142	2730	11	3955
Uranium (dissolved)	µg/L	7	1,142		0		789J	1	789	1360J	1	1360	676J	2	723	138J	2	121	4160J	1	4160
pH	SU	26	6.2	5	2	5.5	6.08	4	6.1	5.83	5	6	6.01	6	6.1	6.03	4	6.8	6.05	5	6.1
Temperature	°C	26	10.6	11.41	2	11.2	9.77	4	10.3	10.62	5	10.5	10.5	6	10.5	11.28	4	10.7	9.94	5	10.7

Notes:

1. All data collected between 2005 and 2012.

Table 3
Field Media Testing and Groundwater Monitoring Schedule
Nuclear Metals Superfund Site
Concord, MA

Sample	Well	Sample Week	Description	Total Samples	U, Fe, Ca, K, Na (total) EPA Method 6020A	U, Fe, Ca, K, Na (dissolved) EPA Method 6020A	V (total) EPA Method 6010	Phosphorous (total) EPA Method 365.4	TIC & TOC EPA Method 9060	Comment
Passive Deployments										
HBPZ2R_P0d_MMDDYYYY	HBPZ-2R	1	Passive day 0	5	X	X	X	X	X	Install temporary pump and perform standard low flow sampling
HBPZ2R_P28d_MMDDYYYY		4	Passive day 28	5	X	X	X	X	X	
HBPZ2R_P84d_MMDDYYYY		12	Passive day 84	5	X	X	X	X	X	
MWS16_P0d_MMDDYYYY	MW-S16	1	Passive day 0	5	X	X	X	X	X	
MWS16_P28d_MMDDYYYY		4	Passive day 28	5	X	X	X	X	X	
MWS16_P84d_MMDDYYYY		12	Passive day 84	5	X	X	X	X	X	
Active Deployments										
MWS24_A0din_MMDDYYYY	MW-S24	1	Active day 0 - influent	4	X	X		X	X	In-line sampling & measurement of field parameters (pH, ORP, cond., DO, T, turbidity)
MWS24_A0deff_MMDDYYYY			Active day 0 - effluent	4	X	X		X	X	
MWS24_A1din_MMDDYYYY			Active day 1 - influent	4	X	X		X	X	
MWS24_A1def_MMDDYYYY			Active day 1 - effluent	4	X	X		X	X	
MWS24_A3din_MMDDYYYY			Active day 3 - influent	1	X					
MWS24_A3def_MMDDYYYY			Active day 3 - effluent	0						
MWS24_A5din_MMDDYYYY			Active day 5 - influent	1	X					
MWS24_A5def_MMDDYYYY			Active day 5 - effluent	1	X					
MWS24_A7din_MMDDYYYY			Active day 7 - influent	8	DUP	DUP		DUP	DUP	
MWS24_A7def_MMDDYYYY			Active day 7 - effluent	4	X	X		X	X	
MWS24_A14din_MMDDYYYY		2	Active day 14 -influent	1	X					Sample in-line & remove media (1)
MWS24_A14def_MMDDYYYY			Active day 14 -effluent	0						
MWS24_A21din_MMDDYYYY		3	Active day 21 - influent	1	X					Sample in-line & remove media (1)
MWS24_A21def_MMDDYYYY			Active day 21 - effluent	0						
MWS24_A28din_MMDDYYYY		4	Active day 28 - influent	4	X	X		X	X	Sample in-line & remove media (1)
MWS24_A28def_MMDDYYYY			Active day 28 - effluent	4	X	X		X	X	
MWS24_A42din_MMDDYYYY		6	Active day 42 - influent	1	X					In-line sampling & measurement of field parameters (pH, ORP, cond., DO, T, turbidity)
MWS24_A42def_MMDDYYYY			Active day 42 - effluent	0						
MWS24_A56din_MMDDYYYY		8	Active day 56 -influent	1	X					In-line sampling & measurement of field parameters (pH, ORP, cond., DO, T, turbidity)
MWS24_A56def_MMDDYYYY			Active day 56 -effluent	0						
MWS24_A70din_MMDDYYYY		10	Active day 70 - influent	8	DUP	DUP		DUP	DUP	Sample in-line & remove media (1)
MWS24_A70def_MMDDYYYY			Active day 70 - effluent	0						
MWS24_A84din_MMDDYYYY		12	Active day 84 -influent	4	X	X		X	X	Sample in-line & remove media (2)
MWS24_A84def_MMDDYYYY			Active day 84 -effluent	4	X	X		X	X	
MW8A_A0din_MMDDYYYY	MW-8A	1	Active day 0 - influent	4	X	X		X	X	Sample in-line & measure field parameters
MW8A_A0deff_MMDDYYYY			Active day 0 - effluent	4	X	X		X	X	
MW8A_A28din_MMDDYYYY		4	Active day 28 - influent	4	X	X		X	X	Sample in-line & remove media (1)
MW8A_A28def_MMDDYYYY			Active day 28 - effluent	4	X	X		X	X	
MW8A_A84din_MMDDYYYY		12	Active day 84 - influent	4	X	X		X	X	Sample in-line & remove media (2)
MW8A_A84def_MMDDYYYY			Active day 84 - effluent	4	X	X		X	X	

Table 3
Field Media Testing and Groundwater Monitoring Schedule
 Nuclear Metals Superfund Site
 Concord, MA

Sample	Well	Sample Week	Description	Total Samples	U, Fe, Ca, K, Na (total) EPA Method 6020A	U, Fe, Ca, K, Na (dissolved) EPA Method 6020A	V (total) EPA Method 6010	Phosphorous (total) EPA Method 365.4	TIC & TOC EPA Method 9060	Comment
Blanks										
Blank_MMDDYYYY	-		Equipment Blank day 0	4	X	X		X	X	-
Blank_MMDDYYYY			Equipment Blank day 28	4	X	X		X	X	
TOTAL				121	34	27	6	27	27	

Notes:

- Groundwater analytical requirements include:
 - Field parameters (pH, ORP, conductivity, DO, temperature, and turbidity) by EPA Low Flow methods.
 - Total and Dissolved Metals and Cations by EPA Method 6020A for uranium, iron, calcium, potassium, sodium, and EPA Method 6010 for vanadium. Sample volumes of 500 mL will be collected and preserved with nitric acid for these analytes.
 - Total phosphorous by EPA Method 365.4 and DIC and TOC by EPA Method 8260. Samples will be stored in amber glass vials with a sulfuric acid preservative.
- A 0.2mm filter will be used for dissolved sample collection.
- The QAPP calls for duplicate groundwater samples at a rate of 1 per 20 for each analyte and an equipment blank at a rate of once for each analyte per sampling round. Because this work includes numerous sampling rounds at only four locations, an equipment blank per sampling event is not practical. We will collect one equipment blank at the beginning of the testing procedures and then another at week four.
- Groundwater samples will be analyzed by GEL Analytical Laboratories in Charleston, SC.

Table 4
Soil Lithologies in Wellscreen Intervals in Uranium Plume
 Nuclear Metals Superfund Site
 Concord, MA

Well	Screen Interval (ft)	Lithology Interval (ft)	Lithologic Description	Stratum
GZW-7-1	68-78	69-71	Dense, yellow-brown, medium to coarse SAND, some(+) GRAVEL (schist and quartz) (wet)	Fine-Medium SAND
		74-76	Dense, light brown-brown, medium to coarse SAND and (-) fine GRAVEL, trace SILT	Medium-Coarse SAND
HBPZ-2R	36-51	35-39	Fine to medium SAND. Little Fine to Medium GRAVEL	Fine-Medium SAND
		39-41	Brown, fine to coarse SAND, some fine to medium GRAVEL, trace(-) SILT	Fine-Coarse SAND & some GRAVEL
		41-43	Brown, fine to coarse SAND, some fine to medium GRAVEL, trace(-) SILT	Fine-Coarse SAND & some GRAVEL
		44-46	Brown, fine to coarse SAND, little fine to medium GRAVEL, trace(-) SILT	Fine-Coarse SAND
		46-48	Black, gray and white COBBLE (stuck in tip).	COBBLE
		48-50	Brown, fine to coarse SAND and fine to coarse GRAVEL, trace(-) SILT	Fine-Coarse SAND & some GRAVEL
MW-8A	60-70	60-62	Wet, very dense, brown, fine to medium SAND, trace(+) fine to medium GRAVEL, trace (-) SILT	Fine-Coarse SAND
		65-67	Very dense, brown, fine to medium SAND, trace(+) fine to medium GRAVEL, trace(-) SILT	Fine-Coarse SAND
MW-S16	69-79	69-75.8	light olive brown (2.5Y 5/4), wet, m. dense, SP SAND, fine sand with 30% medium sand and 10% very fine sand	Fine-Medium SAND
		75.8-77	light olive brown (2.5Y 5/4), wet, m. dense, SW SAND and GRAVEL, medium sand with 20% coarse sand and 15% fine gravel	Medium-Coarse SAND & Fine GRAVEL
		77-79	light olive brown (2.5Y 5/4), wet, m. dense, SW SAND and GRAVEL, medium sand with 30% coarse sand, 10% fine to medium gravel and 10% coarse gravel/cobbles	SAND to GRAVEL
MW-S24	54-64	54-56	Yellowish brown (10YR 5/6) wet, loose SW SAND and GRAVEL; medium sand with 5% sub-rounded large gravel and <5% angular fine gravel. Stratified with darker oxidized layers.	Medium SAND & some GRAVEL
		57-61	Light olive brown (2.5Y 5/4) wet, m. dense SW SAND and GRAVEL; medium sand with 20% fine to medium sub angular gravel and 10% coarse sand, 5% SILT	Medium SAND & some GRAVEL
		61-63	Light olive brown (2.5Y 5/4) wet, m. dense SW SAND and GRAVEL; medium sand with 30-35% medium to coarse sub rounded and sub angular GRAVEL and 5-10% SILT	Medium SAND & some GRAVEL & SILT
		63-65	Light olive brown (2.5Y 5/4) wet, m. dense SW SAND and SILT; medium sand with 10% fine SAND and 1" layer of brown SILT	Fine-Medium SAND
MW-SO2	57-67	56.5-58.5	Light olive gray, moist, dense SP SAND, medium sand lens (1" thick) at 0.5' and 1.8', good cleavage	Medium SAND
		58.5-60.5	Light olive gray, dense, SP SAND, very fine, massive, medium sand lens at 1.8'	Very Fine SAND
		60.5-61.4	Light olive gray, dense, SP SAND, very fine	Fine SAND
		61.4-61.6	Light olive gray, wet, dense, SP SAND, coarse	Coarse SAND
		61.6-61.8	Light olive gray, dense, SP SAND, very fine, massive	Very Fine SAND
		61.8-62.5	SP SAND, GRAVEL, well-sorted, 5% fine gravel, 2% cobble, oxidized at 1.3-1.4'	SAND to GRAVEL
		62.5-63.9	Tan/brown wet, SP SAND, coarse	Coarse SAND
		63.9-64.5	Light olive SP SAND, medium to fine	Fine-Medium SAND
		64.5-66.5	Gray olive, wet, SP/SW SAND, coarse > 20% medium to fine	Medium-Coarse SAND
MW-T24	86-90	85-87	Light olive brown (2.5Y 5/6), wet, medium dense SP SAND, medium to fine SAND with 10% very fine SAND. Discrete interval.	Fine-Medium SAND
		87-87.3	Light olive brown (2.5Y 5/6), wet, medium dense, SP SAND, fine sand with 10% very fine SAND	Fine SAND
		87.3-91	Dark greenish gray (10G 4/1), moist, medium stiff SILT, sandy TILL: SILT with 15% fine to medium sub-rounded GRAVEL and 10-15% medium to coarse SAND	TILL: SILT, Fine Medium GRAVEL, Medium-Coarse SAND
HB-12	45-55	45-47	Wet, very dense, brown, fine to coarse SAND, trace(-) GRAVEL	Fine-Coarse SAND
		47-48		Fine-Coarse SAND
		48-50		Gravelly Fine-Coarse SAND
		50-52	Wet, very dense, brown, fine to coarse SAND, little GRAVEL	Gravelly Fine-Coarse SAND
		52-53		Gravelly Fine-Coarse SAND
		53-55		Fine-Coarse SAND

Notes:

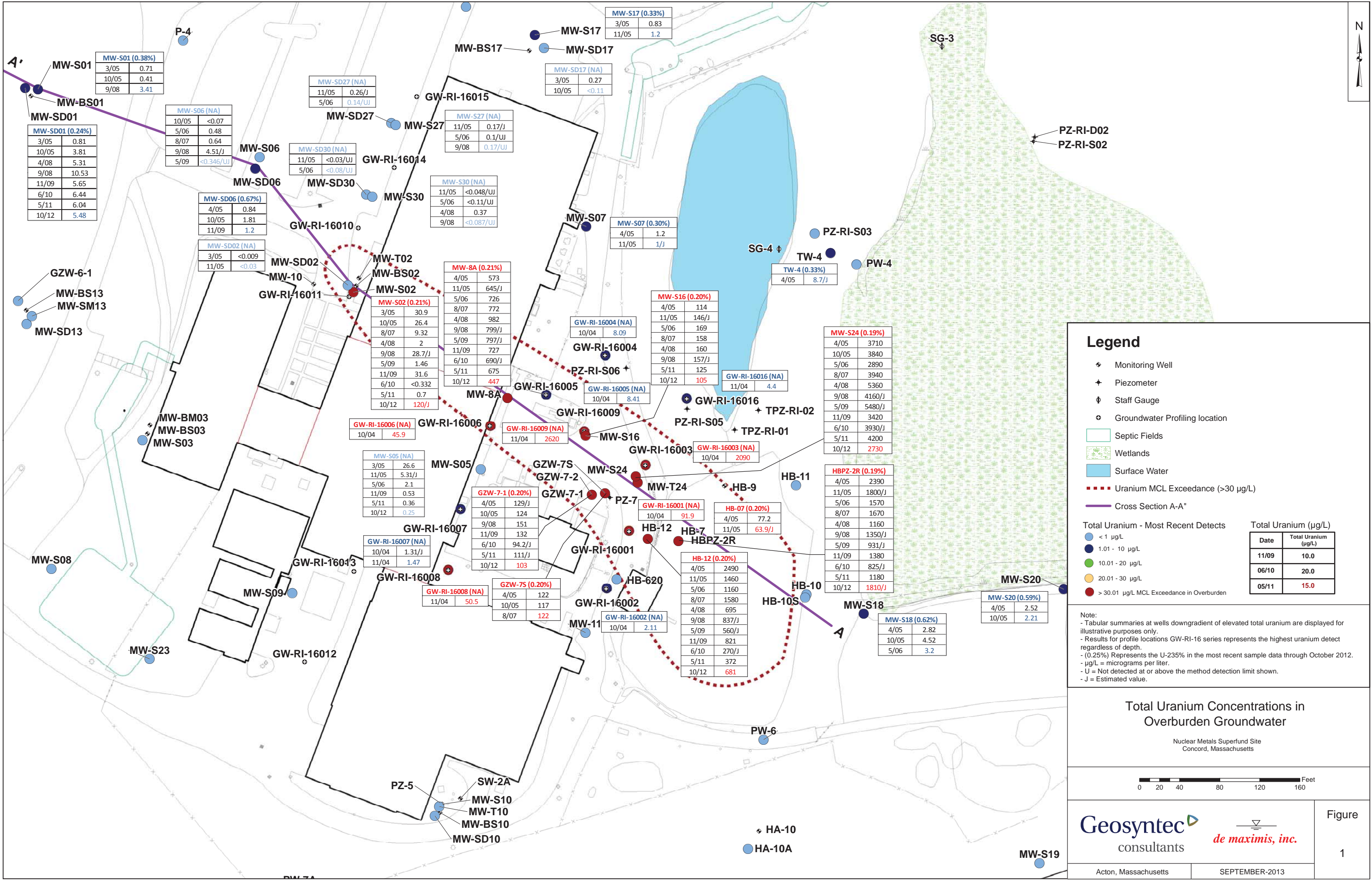
1. Lithologies determined from boring logs generated before and during Remedial Investigation.

Table 5
Proposed Schedule for Media Testing
Nuclear Metals Superfund Site
Concord, MA

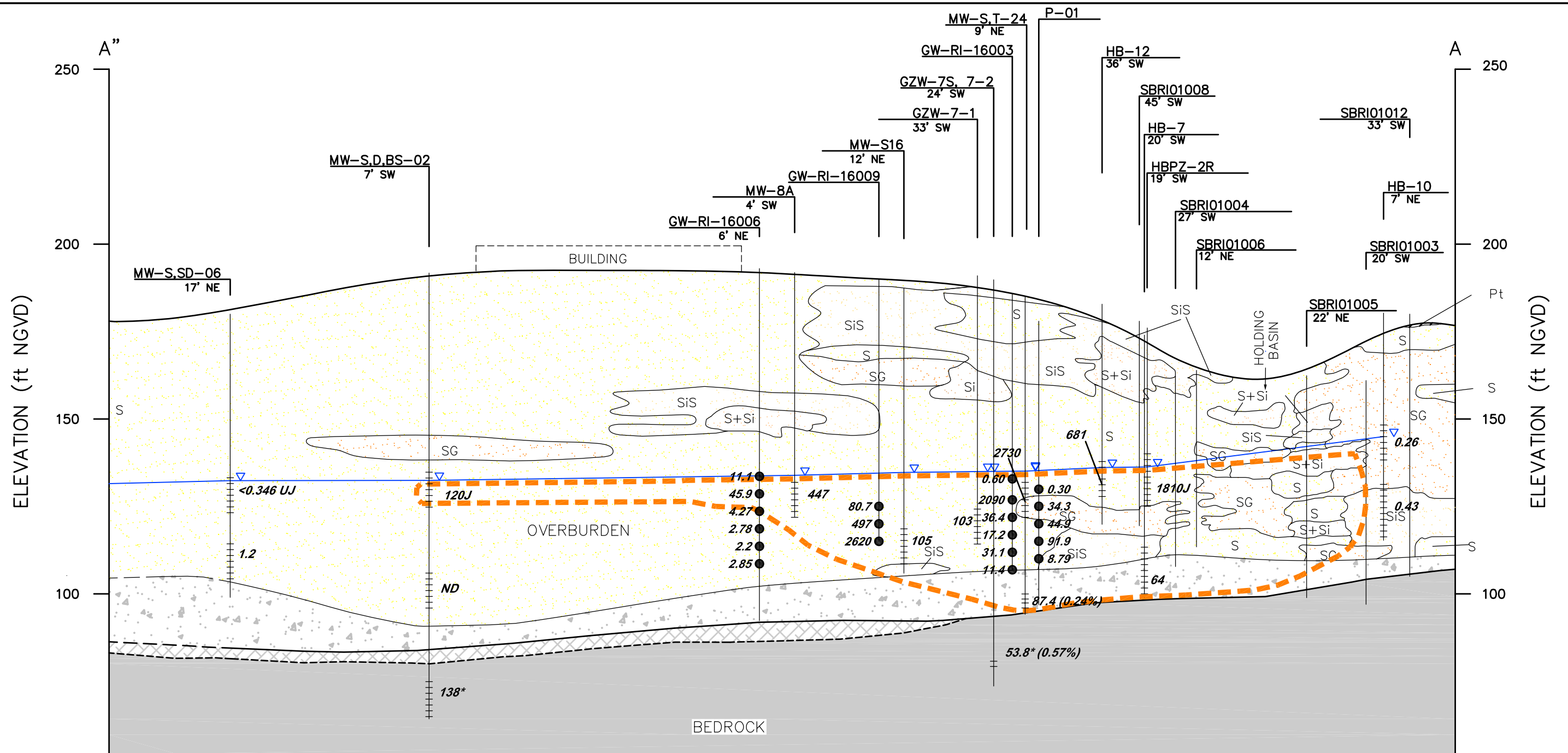
Geosyntec Consultants

	Nov-13	Dec-13	Jan-14	Feb-14	Mar-14	Apr-14	May-14	Jun-14	Jul-14	Aug-14
Acquire PIMS/Synthesize Media										
Media Characterization										
Field Deployment										
Groundwater Sampling										
Field Deployed Media Extraction (30 and 90 days)										
Laboratory Column Studies (laboratory media)										
Leaching Columns (deployed & laboratory media)										
Laboratory Analysis of Field Media										
Proof-of-Concept Technical Memorandum										
Final Report										

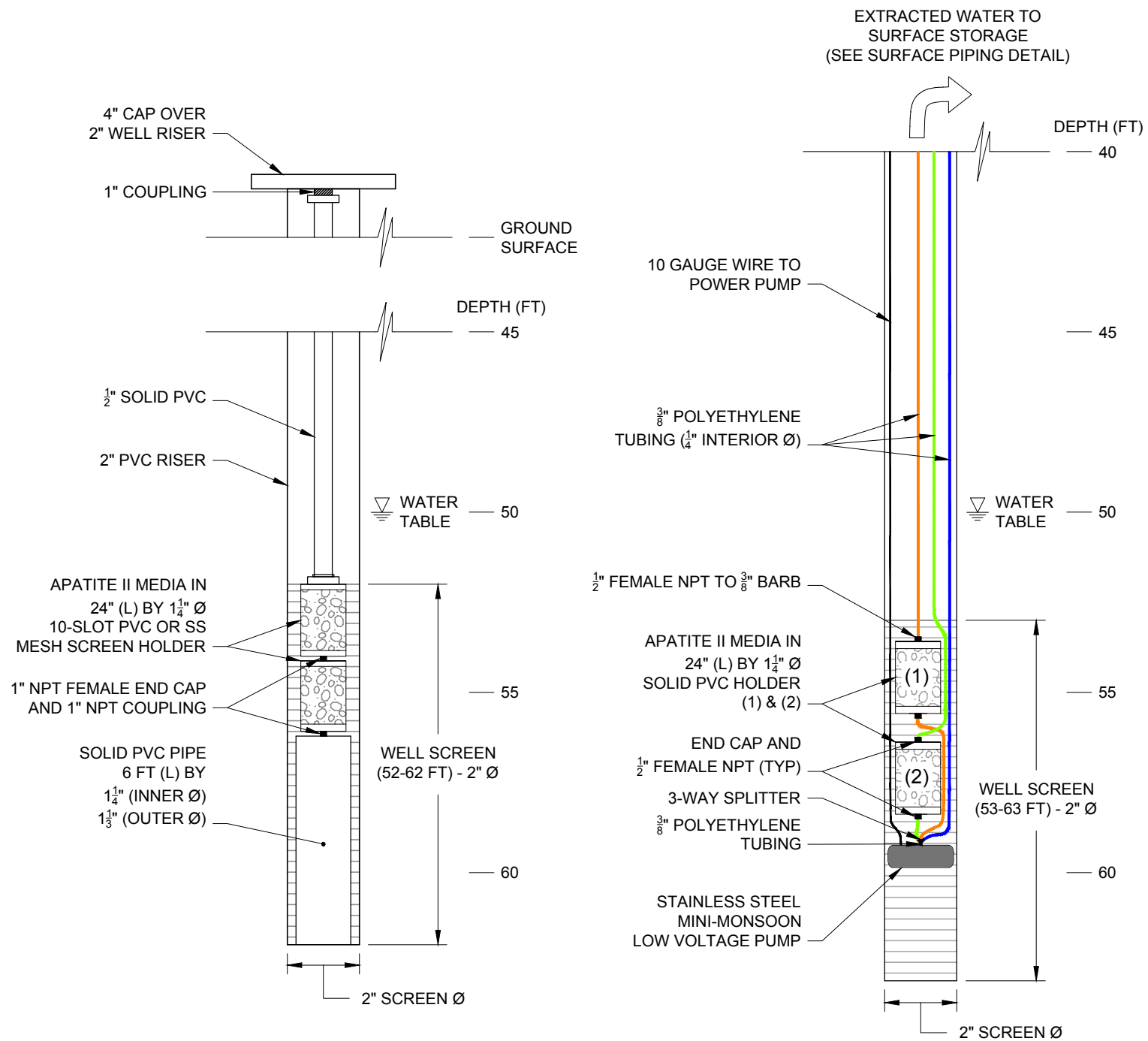
FIGURES



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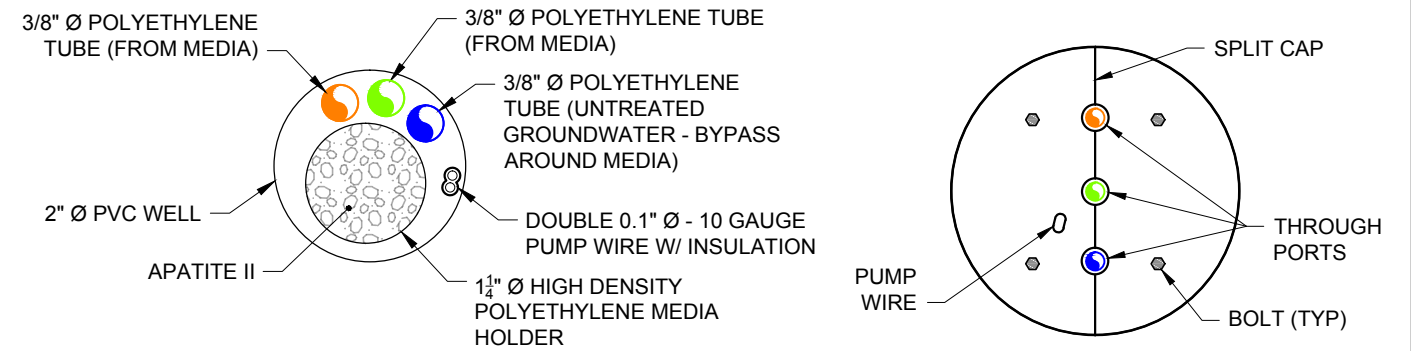


I:_CADD (PROJECTS)\N\NUCLEAR METALS SUPERFUND SITE\FIGURES\F001



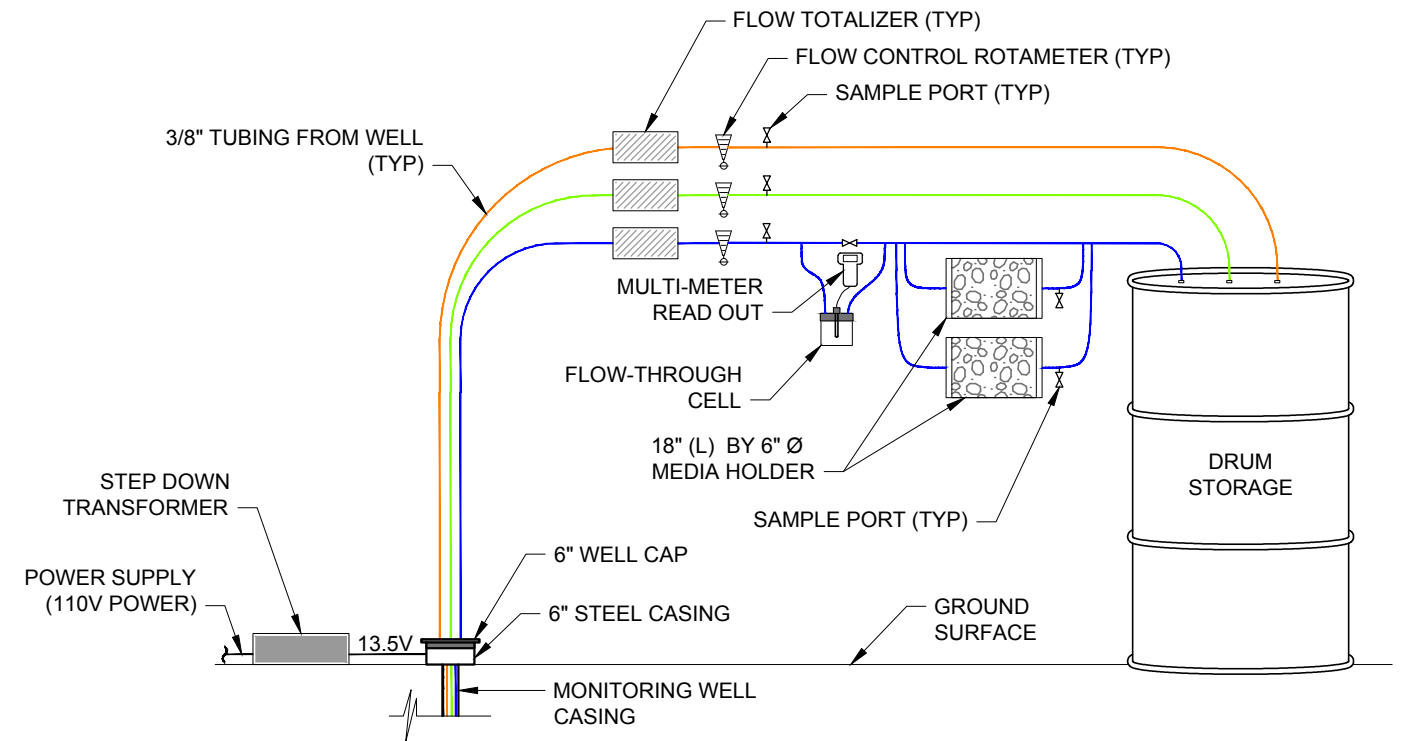
DETAIL (TYPICAL)
IN-WELL PASSIVE COLUMN
TEST APPARATUS
SCALE: 1" = 4" HORIZ, 1"=4' VERT

DETAIL (TYPICAL)
IN-WELL ACTIVE COLUMN
TEST APPARATUS
SCALE: 1" = 4" HORIZ, 1"=4' VERT



CROSS-SECTION
IN-WELL
SCALE: 1" = 2"

DETAIL
3-HOLE WELL CAP
SCALE: 1" = 4"



DETAIL
SURFACE PIPING AND INSTRUMENTATION FOR
ACTIVE COLUMN APPARATUS
SCALE: NTS

NOTE:

1. APATITE II IS A PROPRIETARY MEDIA DEVELOPED BY PIMS NW, INC.

IN-WELL COLUMN TESTING DESIGN
NUCLEAR METALS SUPERFUND SITE
CONCORD, MA

Geosyntec
consultants

Acton , MA

SEPTEMBER 2013

Figure
3

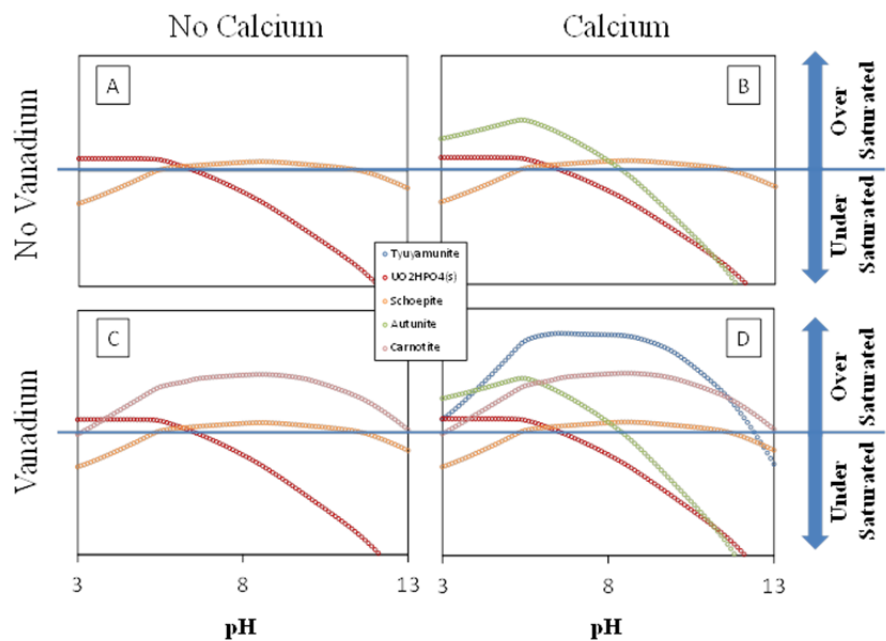


Figure 4. Thermodynamic modeling of uranium(VI) solid phase saturation indices under conditions of (A) uranyl and phosphate (B) calcium, uranyl and phosphate (C) vanadate, uranyl and phosphate and (D) calcium, vanadate, uranyl and phosphate (*SMU Water and Soil Chemistry Laboratory*).

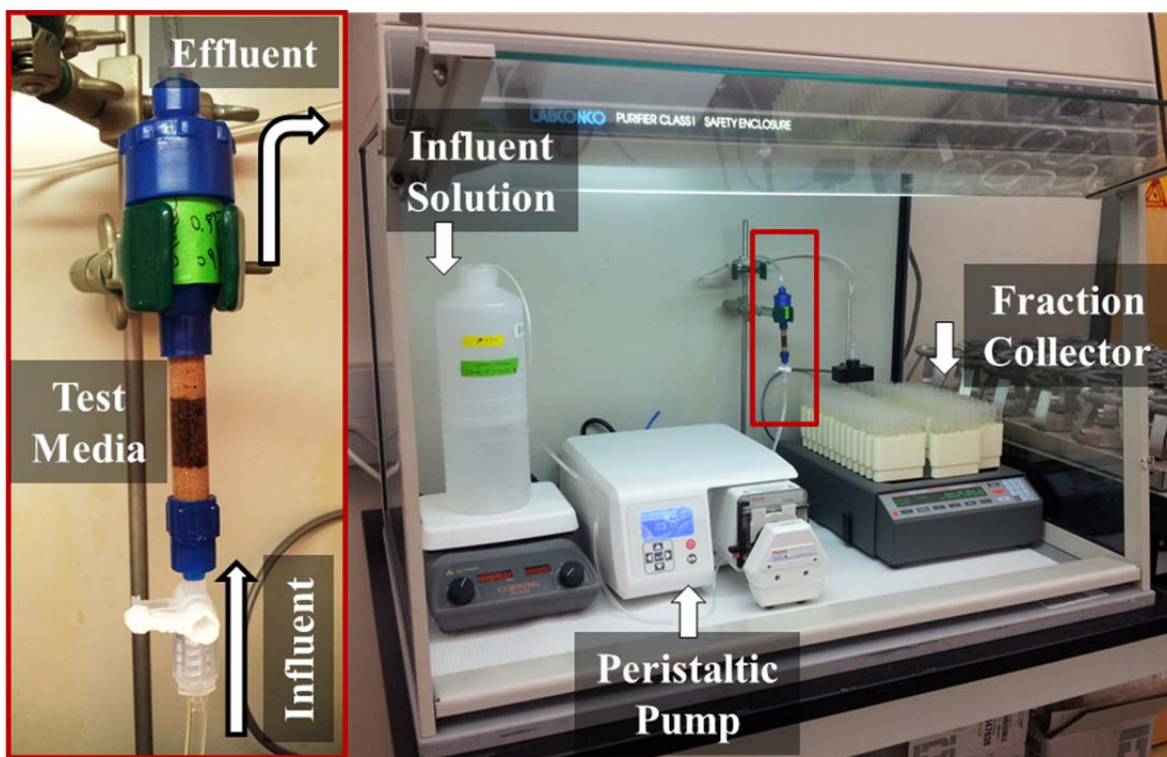


Figure 5. Example laboratory column experimental set-up (*SMU Water and Soil Chemistry Laboratory*) .

APPENDIX A

Low Flow Sampling Procedures and Field Forms

U.S. ENVIRONMENTAL PROTECTION AGENCY REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

Quality Assurance Unit
U.S. Environmental Protection Agency – Region 1
11 Technology Drive
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1/19/10
Date

Approved by: Gerard Sotolongo
(Gerard Sotolongo, Quality Assurance Unit)

1-19-10
Date

Revision Page

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TABLE OF CONTENTS	Page
USE OF TERMS	4
SCOPE & APPLICATION	5
BACKGROUND FOR IMPLEMENTATION	6
HEALTH & SAFETY	7
CAUTIONS	7
PERSONNEL QUALIFICATIONS	9
EQUIPMENT AND SUPPLIES	9
EQUIPMENT/INSTRUMENT CALIBRATION	13
PRELIMINARY SITE ACTIVITIES	13
PURGING AND SAMPLING PROCEDURE	14
DECONTAMINATION	19
FIELD QUALITY CONTROL	21
FIELD LOGBOOK	21
DATA REPORT	22
REFERENCES	22
APPENDIX A PERISTALTIC PUMPS	24
APPENDIX B SUMMARY OF SAMPLING INSTRUCTIONS	25
LOW-FLOW SETUP DIAGRAM	29
APPENDIX C EXAMPLE WELL PURGING FORM	30

USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Potentiometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convection cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or Teflon are preferred. Note: if extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a "best practice". For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

Teflon or Teflon-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Note: if tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A "T" connector coupled with a valve is connected between the pump's tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, January 19, 2010, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed lay out sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs).

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each

sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). All during the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods (e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size ($0.45\ \mu\text{m}$ is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and then following sampling of each well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

REFERENCES

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U.S. Environmental Protection Agency, Region 1, *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, January 19, 2010 or latest version.

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APPENDIX A PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could effect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).
2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.
3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.
4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.
5. Measure water level and record this information.
6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or colored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or colored water is usually from the well being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

Wait for the well to recharge to a sufficient volume so samples can be collected. This may take awhile (pump maybe removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note,

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record that the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

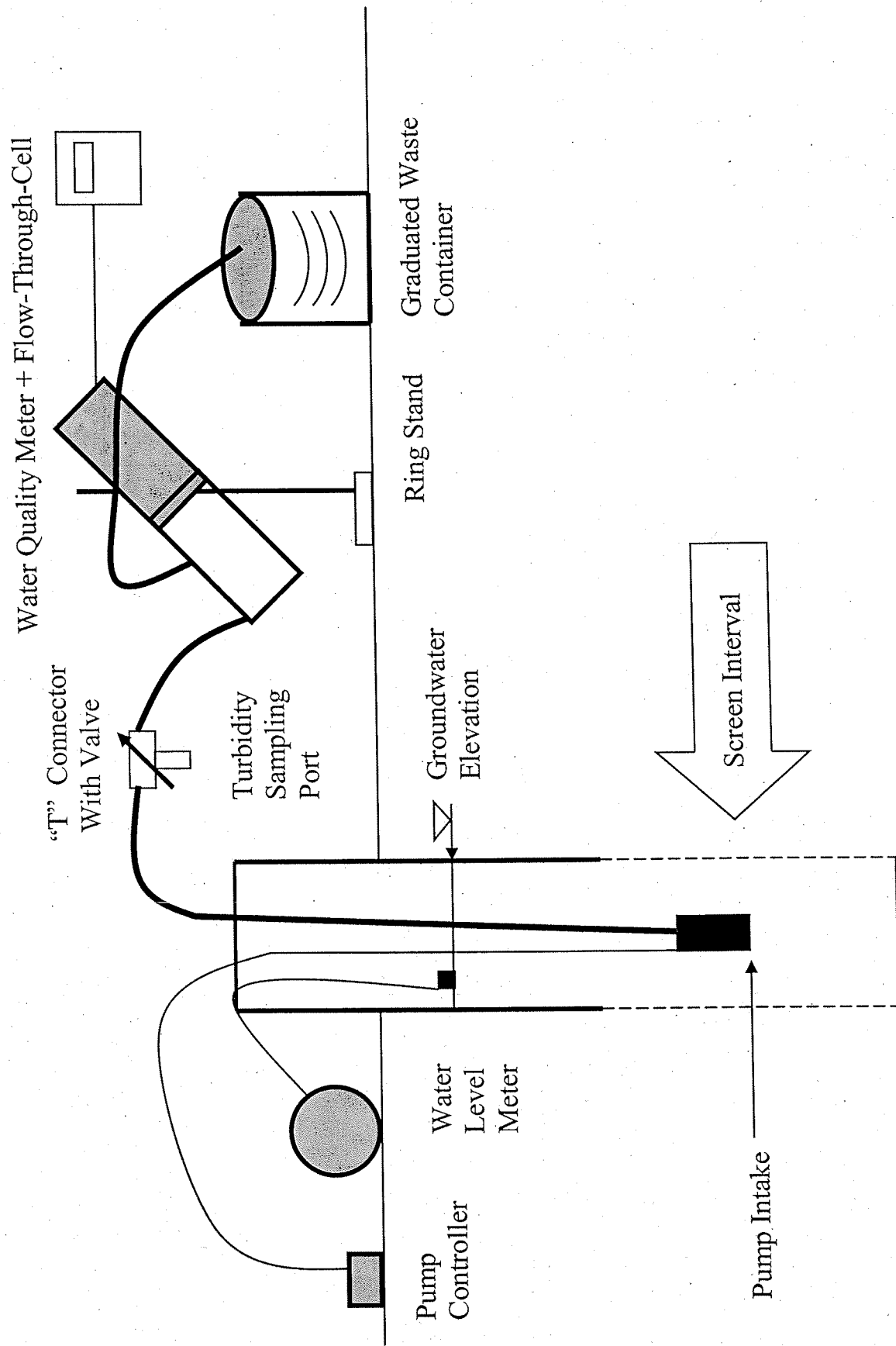
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



EXAMPLE (Minimum Requirements)

WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

[illegible]

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. μ Siemens per cm (same as μ mhos/cm) at 25 °C.
3. Oxidation reduction potential (ORP)